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Polyarylacetylene Resin Composites

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Polyarylacetylene (PAA), a highly cross-linked aromatic polymer that has a char yield of 90% when pyrolyzed in an inert environment, has been investigated in a major research and development program designed to realize the potential benefits of this material in space system components. The recent achievements of the Materials Sciences Laboratory in developing the processing techniques needed to fabricate carbon-fiber-reinforced PAA composites are outlined. Large, thick composites were fabricated and were demonstrated in various practical applications. Applications include dimensionally stable space system composite structures, ablative insulators for solid-rocket-motor nozzles and exit cones, and precursors for relatively low-cost carbon-carbon composites.

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PREFACE

An ongoing team of MSL staff members has been investigating polyarylacetylene (PAA). Current participants include W. T. Barry, A. Y. Craig, G. C. Denault, M. P. Easton, C. A. Gaulin, T. W. Giants, H. G. Hoppe, H. A. Katzman, R. W. Kobayashi, J. J. Mallon, J. A. Noblet, G. S. Rellick, D. C. Robinson, P. M. Sheaffer, R. A. Shenk, S. L. Zacharius, and R. J. Zaldivar.

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I. INTRODUCTION

Polyarylacetylene (PAA) is a highly cross-linked aromatic polymer that contains only carbon and hydrogen. Its principal advantage is that only about 10 wt% is volatilized when the polymer is heated to high temperatures in an inert environment; the remaining 90 wt% is carbon char. Realizing the benefit of this high char yield in high-performance space system components has required a major research and development program.

As part of this long-term, continuing program, the Materials Sciences Laboratory (MSL) of The Aerospace Corporation is currently investigating PAA for its potential use in dimensionally stable space system composite structures, as a superior ablative insulator for solid-rocket-motor nozzles and exit cones, and as a precursor for low-cost carbon-carbon (C-C) composite structures.

This report presents a brief history of PAA resins (Section II) and the materials and processing challenges that were met to achieve an engineering material (Section III). Space system applications of PAA composites are then described (Section IV). Finally, the recent accomplishments of the MSL development program are summarized in Section V.

II. A BRIEF HISTORY OF POLYARYLACETYLENE RESINS

Polyarylacetylene was first synthesized in the late 1950s at the GE Research Laboratory as part of a broad search for polymers with high char yield.[1] During cure, that early formulation underwent severe shrinkage and released large amounts of heat (exothermic reaction). Such processing difficulties precluded the practical application of that material. In the early 1960s, techniques for reducing both the exotherm and extent of shrinkage during polymer curing were demonstrated at Cyanamid Corp.[2] However, those techniques were not applied to PAA until the early 1970s, when Hercules patented a process for the production of PAA from diethynylbenzene (DEB) monomer for use as a high-temperature molding compound (designated HA 43 by Hercules).[3] HA 43 was made available in pilot-plant quantities for evaluation by the technical community. The Aerospace Corporation used it to produce a carbon-fabric-reinforced ablative thermal protection system. Although they ablated satisfactorily, the composites were brittle and had poor structural integrity. Further research at Aerospace demonstrated HA 43 to be a suitable carbon matrix precursor for special x-ray-absorbing carbon-carbon nuclear shields. The Defense Nuclear Agency (DNA) then funded MSL to direct a program to construct and evaluate thin-walled x-ray-absorbing C-C composites. Samples of those composites were evaluated in aboveground and underground test environments and exhibited excellent survivability.[4]

In the early 1980s, growing requirements for laser countermeasures stimulated the current PAA research program at Aerospace. MSL found that the Hercules-patented molding material lacked the plasticity to make plies of material impregnated with resin (prepreg). The fabrication of large parts by standard techniques was thus prohibited. Although the Hercules patent teaches that addition of low molecular weight materials can provide sufficient plasticity for effective molding of the high molecular weight prepolymer at reasonable pressures, research in MSL showed that such low molecular weight materials are not incorporated into the final polymer.

Consequently, a structurally poor composite with low strain capability (brittleness) and reduced char yield is produced.

The Materials Sciences Laboratory program was designed to correct these deficiencies. They were overcome by the development of a controllable, low-temperature prepolymerization technique that produces a soluble, easy-to-process prepolymer. We demonstrated that polymer-chain modifications to improve ductility and toughness can be incorporated during prepolymer synthesis, resulting in composites with high char yield and good mechanical properties.[5] Detailed accomplishments in MSL's PAA material and process development are discussed in the following sections.

III. MATERIAL AND PROCESS DEVELOPMENTS FOR PAA COMPOSITES

In the MSL technique for fabrication of PAA-based carbon-fiber-reinforced composites, shown in Fig. 1, a solution of meta- and para-DEB monomer is first cyclotrimerized into a low molecular weight prepolymer. In cyclotrimerization, three acetylene groups (from three different monomer molecules) react to form a benzene ring. Carbon fiber tows or woven fabrics are then coated with the prepolymer to produce a prepreg unidirectional tape or woven fabric. This prepreg can be wrapped or wound or stacked and formed into the desired shape by conventional lay-up procedures, and the polymer can be cured under moderate pressure (100-200 psi) at a relatively low temperature (100-150°C). The resin matrix composites can then be used as primary structures or they can be heat-treated to carbonize or graphitize the resin to form a relatively high-density C-C composite.

Processing problems that required solution were (1) characterizing and controlling the purity of the monomer; (2) developing a prepolymer with the proper degree of cyclotrimerization to yield soluble, stable material with the required rheological characteristics; and (3) controlling the processing for prepreg lay-up, consolidation, and curing. Each of the PAA composite fabrication steps is detailed below.

A. MONOMER

All of the DEB monomer used in the early work in MSL was synthesized in small batches in our laboratory. Thus, the size of composite articles that could be fabricated and studied was seriously limited. In 1988, we learned that Hercules had stored about 150 kg of DEB remaining from their production of HA 43 resin. We purchased 50 kg of that material, which enabled us to fabricate and test large-scale composite parts. (A reliable supply of material is expected to become available soon, since Hercules recently announced that they will manufacture both DEB monomer and cyclotrimerized prepolymer.)

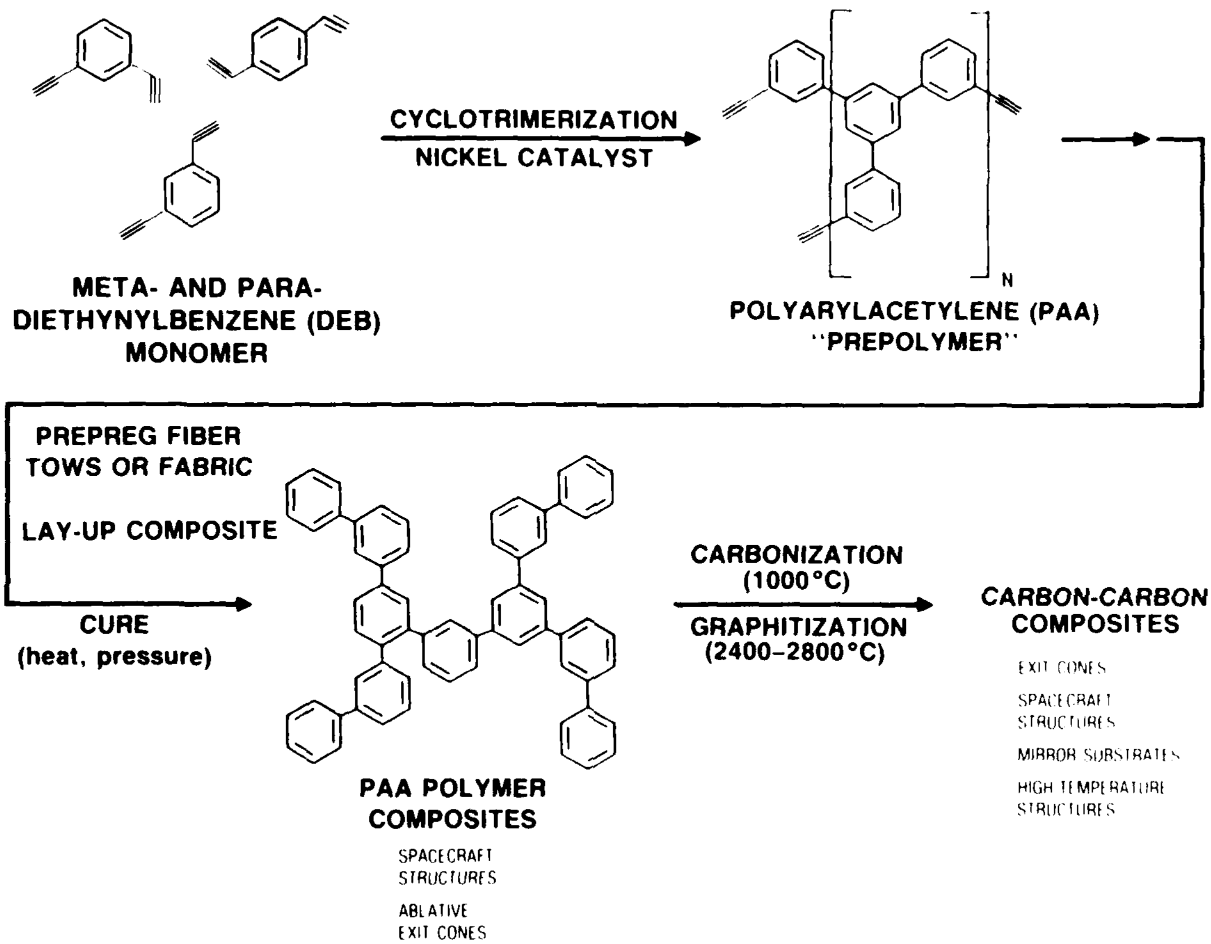

Fig. 1. Schematic of fabrication of polyarylacetylene composites.

Successful fabrication of PAA composite parts requires that the DEB monomer be of high purity so that the formation and subsequent outgassing of volatile by-products is reduced during composite fabrication and use Standard analytical methods and separation techniques were found to be inadequate to characterize monomer purity. However, we were able to demonstrate that monomer isomers and their by-products are readily distinguishable by proton nuclear magnetic resonance (pNMR) spectroscopy. Figures 2a and 2b are NMR spectra of 99% isomerically pure para- and meta-DEB synthesized in MSL. Figure 2c is a spectrum of DEB received from Hercules; the sharp singlets at 3.1 and 3.2 ppm and the region centered at 7.4 ppm in Fig. 2c are predominantly due to a mixture of meta- and para-isomers in a 3:2 ratio. The other resonances below 3 ppm represent impurity by-products (ethyl, vinyl, and ethynyl substituents), which can be quantitatively determined by peak area ratios. Such impurities are undesirable for PAA composite fabrication. We have used pNMR to verify and control the purity of the monomer received from Hercules.

B. PREPOLYMER

The cyclotrimerization step is designed to produce a soluble solid that melts at a temperature lower than the onset of cure. Cyclotrimerization is important because it produces resonance-stable benzene rings that govern both the thermal stability of the system and the high char yield on pyrolysis. It is an exothermic reaction; therefore, part of the heat of polymerization is liberated during this step, resulting in a milder and more controllable exotherm during final cure. In addition, part of the shrinkage that accompanies curing occurs during cyclotrimerization. Successful PAA composite processing requires that the prepolymerization be stopped when the reaction has proceeded to the required point. Too little reaction negates the benefits of the cyclotrimerization step; on the other hand, a reaction permitted to proceed too far forms a prepolymer that is very difficult to process. It is also crucial to prevent the occurrence of a reaction which competes with cyclotrimerization: linear polymerization. Linear polymerization results in a polymer with reduced thermal stability and char yield.

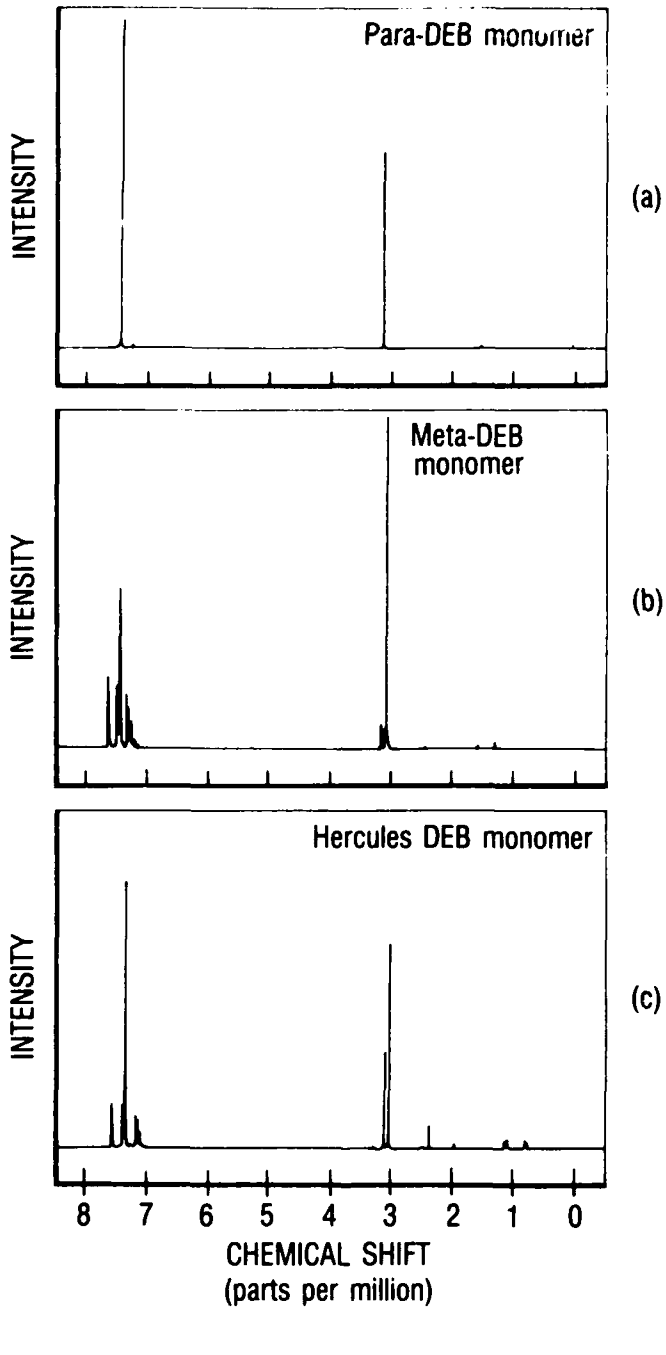

Fig. 2. Proton nuclear magnetic resonance (pNMR) spectra of DEB: spectra of 99% isomerically pure (a) para-DEB and (b) meta-DEB, synthesized in MSL; and (c) spectrum of DEB received from Hercules.

We established that methyl ethyl ketone (MEK) is a better solvent for the cyclotrimerization reaction than the previously used toluene. The use of MEK results in rapid reaction, and its low boiling point enables good process control. In 1989, we demonstrated the use of infrared (IR) spectroscopy to monitor and control the prepolymer reaction. In this technique, the ratio of the intensities of an IR peak associated with the synthesized prepolymer (at 1600 cm^{-1} due to ring-to-ring C-C stretching) to that of an IR peak from the acetylenic groups that are being consumed ($C \equiv H$ stretching at 3256 cm^{-1}), is monitored as the reaction proceeds. The ratio of intensities of these two peaks at various reaction times has been correlated with the processability of the resulting prepolymer, and an optimum ratio has been determined.

The chemical structure of the MSL prepolymer and the possible presence of undesirable linear polymerization were investigated with pNMR spectroscopy. A pNMR spectrum of the prepolymer is shown in Fig. 3. Linear polymerization would be indicated by the presence of olefinic protons in the spectral region between 5.3 and 6.8 ppm. The spectrum shows no detectable peaks in this region, confirming that no linear polymerization has occurred.

C. PREPREG

In the next step of the PAA composite fabrication process, either fiber tows or woven fabric is impregnated with the prepolymer solution and the MEK solvent is evaporated, leaving a prepreg tape or fabric. The handleability of this prepreg is crucial for good composite fabrication: The prepreg must be easy to drape over curved surfaces and be tacky enough to keep the prepreg layers from moving in relationship to each other. The resin must also melt and flow before curing to completely impregnate and consolidate the laminated preform.

We have successfully developed a procedure for controlling drape, tackiness, and flow of the prepreg. Phenylacetylene, a chemical analog of DEB, contains only a single active acetylene group and can be cocyclotrimerized with the DEB. This coprocessing leads to a prepolymer that is less

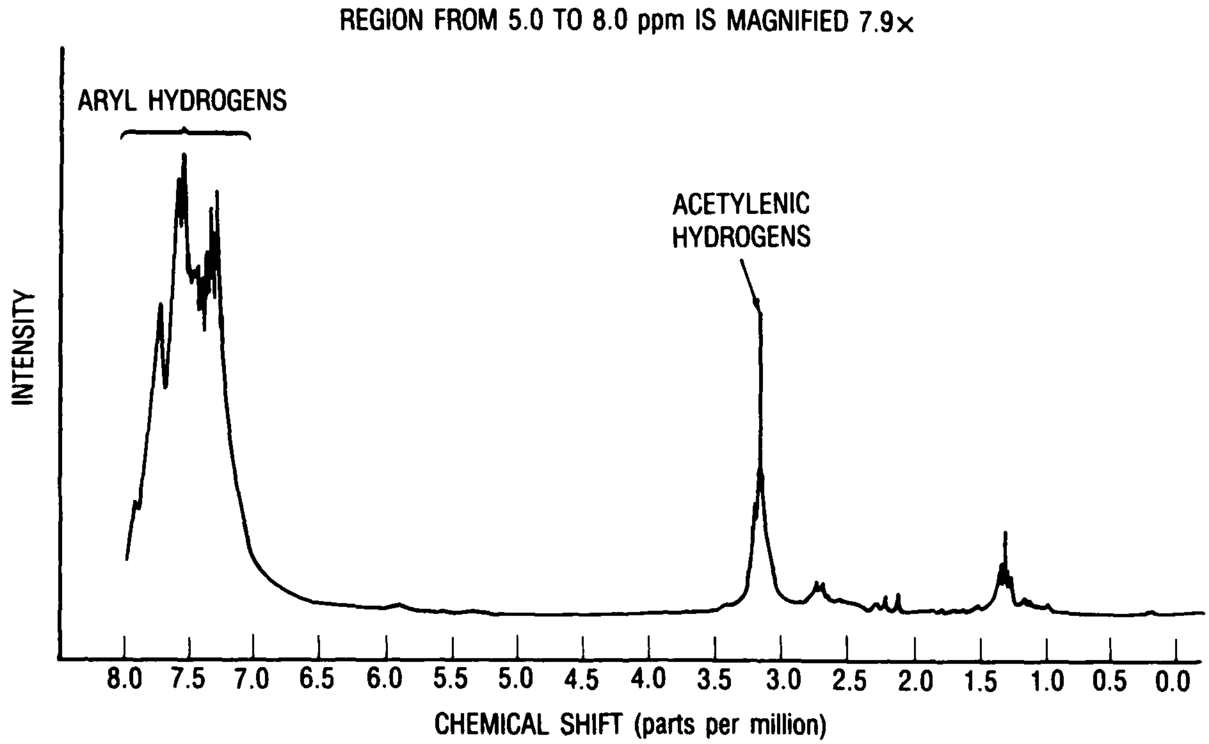

Fig. 3. Proton nuclear magnetic resonance spectrum of PAA prepolymer synthesized in MSL.

cross-linked and, therefore, less viscous when melted. To further control the flow properties, DEB monomer can be blended with the prepolymer, compensating for any monomer or prepolymer variation. We have found that such blending does not decrease the high pyrolysis char yield of the cured resin.

D. CURE

The PAA composite lay-up may be cured by conventional vacuum-bag processing, in an autoclave, hydroclave, or press. No special tooling or apparatus is required.

IV. APPLICATIONS OF PAA MATRIX COMPOSITES

PAA resin matrix composites are being investigated in MSL for several potential applications, including (1) conventional resin matrix composites with ultralow-moisture outgassing characteristics and improved dimensional stability for spacecraft structures, (2) precursors for carbon-carbon composites, and (3) ablative insulators for solid rocket motors.

A. SPACECRAFT STRUCTURES

The main motivation for investigating PAA as a matrix for carbon fibers in spacecraft structural materials is the very low moisture absorption and outgassing of PAA resin. Materials with low moisture absorption are not subject to gradual dimensional change with changes in humidity. Moreover, in space there will be no evolution and condensation of moisture on sensors and mirrors to degrade performance and limit mission life. The graphite-epoxy composites currently used for space structures display excessive moisture absorption because of the hydrophilic nature of epoxy resins. These contain numerous polar functional groups, such as hydroxyls and amino groups, that readily hydrogen-bond with water. In contrast, PAA is hydrophobic, which means that it contains no such polar groups. As a result, it absorbs relatively little moisture, as shown in Fig. 4.

We have determined that the strength and modulus of carbon-fiber-reinforced PAA composites are comparable to those of graphite-epoxy. Mass loss and collected volatile condensable material testing of PAA composites was performed in accordance with ASTM and NASA specifications, and PAA composite outgassing was well below acceptable limits.

B. CARBON-CARBON COMPOSITES

Carbon-carbon composites offer outstanding thermodynamic and dimensional stability over wide temperature ranges and are resistant to laser and nuclear threats. They are used currently in a number of DoD space systems and have replaced previously used monolithic polycrystalline graphite in several solid-rocket-motor throat inserts to provide superior

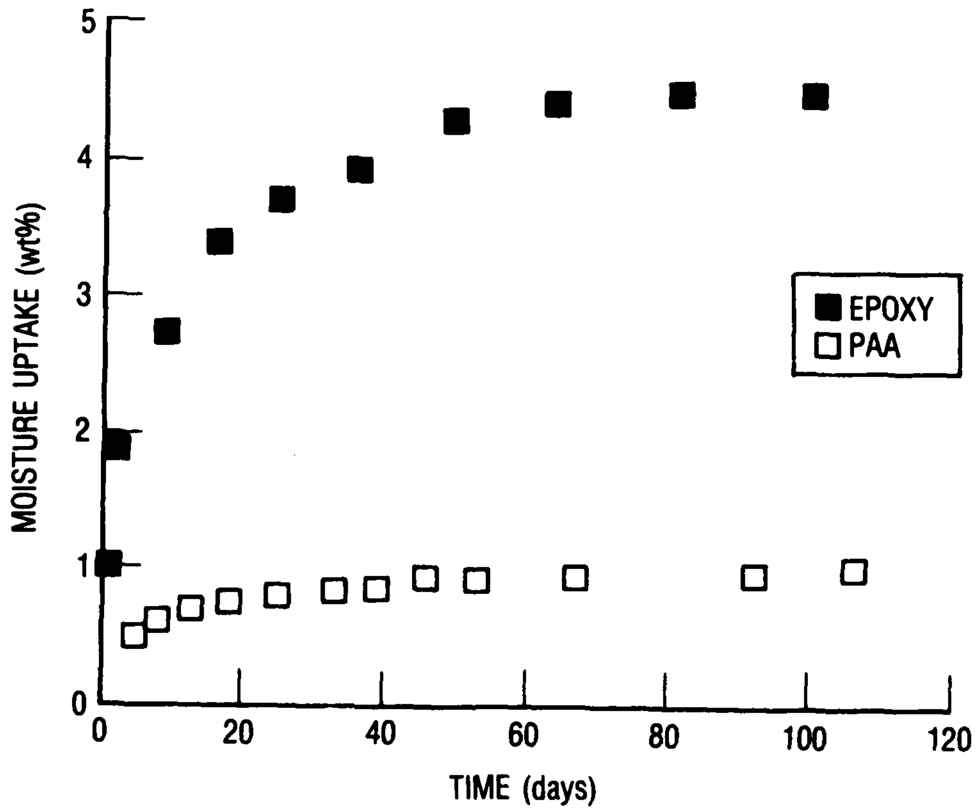

Fig. 4. Moisture uptake by neat resins at 80% relative humidity and 25°C.

reliability. They are in production for lightweight exit cones in ballistic missile boosters and in the IUS, and are under development or consideration for a number of emerging systems. C-C composites are now also being considered for survivable spacecraft structural applications in future systems, such as large space structures and mirror substrates.

The major problem in existing systems is cost. Conventional C-C processing requires four to six months to complete a typical component and achieves the required material density by repeated and lengthy impregnation and high-temperature heat-treatment cycles. Such processing is very costly, and the schedule effect of discrepancies and process mishaps is extreme because of the long time needed to fabricate replacement hardware.

PAA resins are particularly attractive as carbon matrix precursors because their very high carbon yields can eliminate or reduce the multiple impregnation cycles required of current precursors. Figure 5 illustrates the superior char yield of PAA over that of currently used phenolic. The costly multiple impregnation cycles also increase the likelihood of processing failures and contribute to fiber-strength degradation. Relatively low volume shrinkage upon carbonization (Fig. 6) is another attractive feature of PAA resins. Matrix shrinkage is thought to reduce composite strength by introducing surface defects into the fibers.

Carbon-carbon composites containing either carbon fibers or fabric reinforcements utilizing PAA resin as the carbon matrix precursor have been fabricated and evaluated in MSL.[6] They exhibit good mechanical properties and dimensional integrity after carbonization and graphitization, requiring no additional reimpregnation and carbonization cycles to achieve these desirable properties.

C. ABLATIVE COMPONENTS FOR SOLID ROCKET MOTORS

Carbon-phenolic (C-Ph) and graphite-phenolic (Gr-Ph) materials are currently used for ablative and insulative components of booster nozzles and exit cones in Titan, Delta, Shuttle, and MX solid rocket motors (SRMs). Ideally, these materials should exhibit repeatable, predictable, uniform recession rates at the flame surface and should thermally insulate

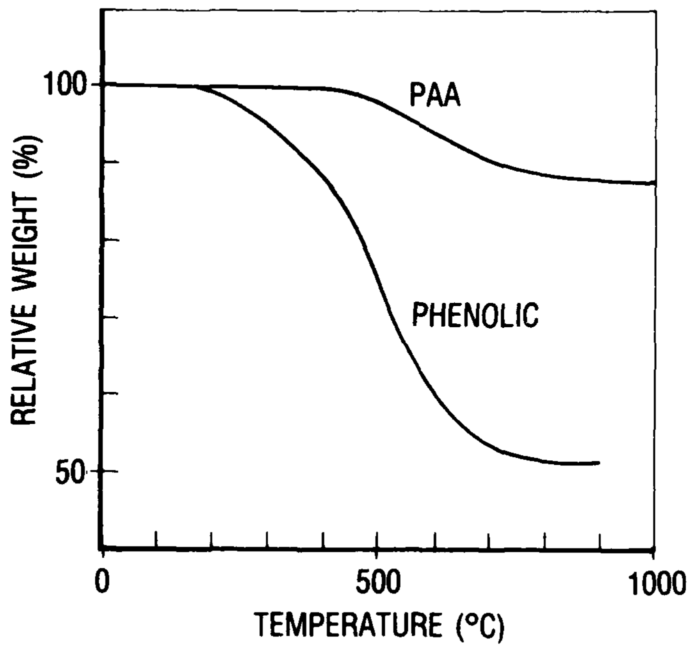

Fig. 5. Weight loss of PAA and phenolic resin as a function of temperature. The weight of material remaining after heating to high temperatures in an inert environment is termed char yield.

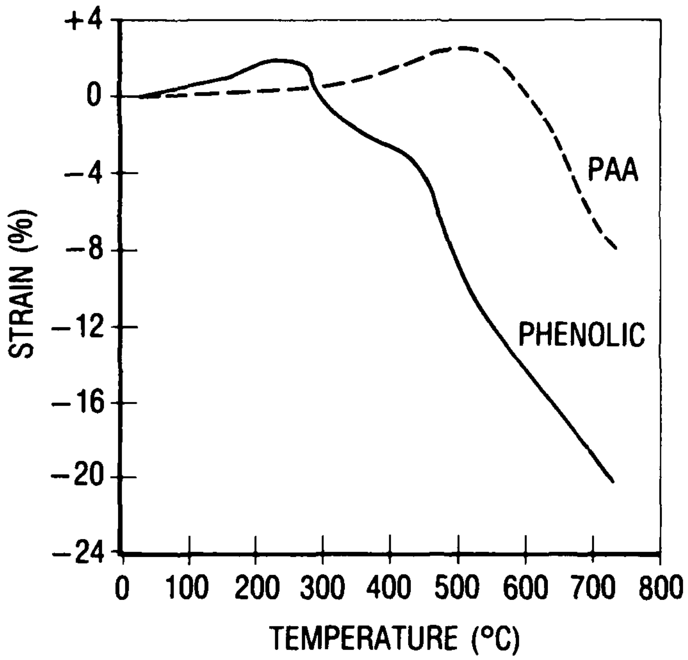

Fig. 6. Pyrolysis shrinkage of neat PAA and phenolic resin. At 750°C, PAA has undergone less than half the shrinkage of the phenolic resin. The positive volume change at lower temperatures is due to swelling caused by polymer degradation.

the backside structure as well. In operation, however, the ablation behavior of phenolic matrix ablative materials has been variable and unstable, posing doubts about the true system reliability. Although no system failures have been attributed to the behavior of these phenolic materials, the more serious incidents have led to costly redesign and recovery efforts.

The best-known example of unstable ablation is the "pocketing" discovered in the C-Ph nozzle components recovered from the STS-8A booster (Fig. 7). Localized deep pockets in a regular array were found. In the worst region, only a few seconds of burn-through life margin remained. NASA conducted a major materials investigation and redesign effort to avoid recurrence, and initiated evaluation of material replacements. However, incidents of less severe pocketing have been reported for subsequent STS flights. More recently, Titan IV static tests revealed unstable ablation in two localized zones opposite the exit cone O-ring seal. Although booster performance was otherwise nominal, unstable ablation eroded the phenolic material as far as the O-ring seal groove, leaving zero margin.

Other examples of unexplained and disturbing incidents of erosion pits, shallow craters, and grooves have accompanied unexpected and unpredictable ablation. Some solid motor test anomalies have also been attributed to the large amounts of evolved pyrolysis gases generated by the phenolic matrix materials. Inadequate management of pyrolysis gas can lead to extremely high pressures within the composite materials. The forces exerted by such pressures exceed the design capability of nozzle components, causing expulsion of gas and buckling of exit cones. In a dramatic example (D5 program), such forces propelled the throat section forward at the end of burn, causing the section to violently strike the chamber nozzle forward dome.

Such problems result from the hydrophilic nature of phenolic resins (Fig. 8), which cure by a condensation reaction that has water as a byproduct and also readily absorb moisture. The absorbed moisture acts as a plasticizer and lowers the interlaminar load-carrying capacity of the

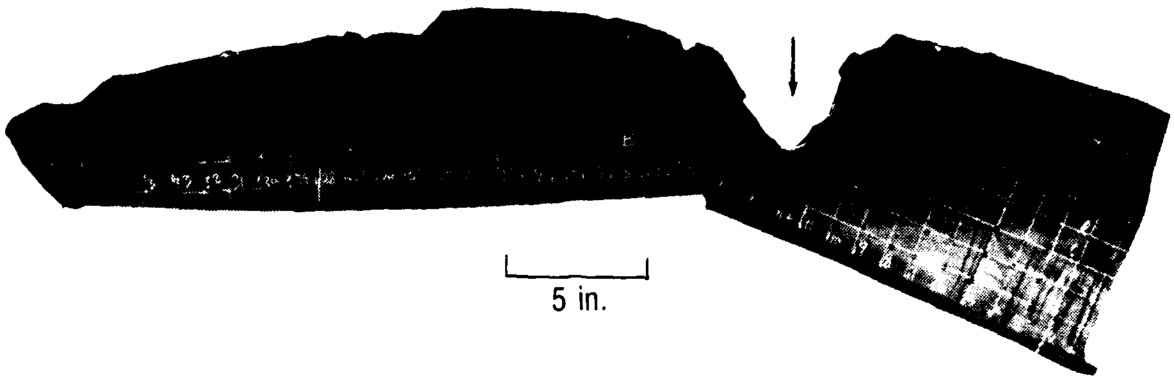

Fig. 7. Photograph of a localized erosion pocket in carbon-phenolic nozzle and throat structure of the STS-8A solid booster, indicating unstable ablation. Arrow indicates worst-case erosion, in which only a few seconds of life remained before burn-through.

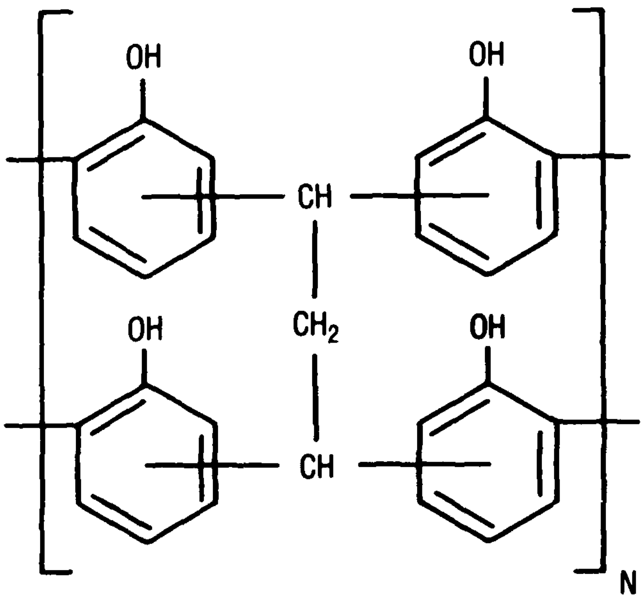

Fig. 8. Chemical structure of phenolic resin.

resin. During ablative heating, the moisture is vaporized and causes pressure to build up within the composite, resulting in an array of internal delaminations (ply lifts) that extends to the outer surface, causing local sloughing and enhanced ablation. That pressure is in addition to the pressure caused by the large amount of volatile hydrocarbons from pyrolysis of the resin itself, which has a char yield of only about 50%. These volatiles can be thermally cracked, resulting in carbon deposition and plugging of gas-diffusion paths, and hence greater internal pressure buildup. Pressure in the pores can also cause tensile failure of the yarns, leading to ejection of chunks of unattached material and localized pockets. Moreover, the residual phenolic char has no useful load-carrying capacity.

In contrast, the hydrophobic PAA resin does not absorb much moisture and cures by an addition reaction that has no by-products. Its char yield of about 90% means that far less volatile material is generated and that minimal shrinkage is associated with pyrolysis. We measured the composition and relative amounts of gases released during pyrolysis of PAA neat resins using gas chromatography mass spectrometry (Fig. 9).[7] The dominant gas evolved by PAA is hydrogen, the amount of which peaks at 800°C. In contrast, the gaseous pyrolysis products of phenolic resin are dominated by high molecular weight hydrocarbons, as well as oxygen-containing hydrocarbons, that peak at the lower temperature of 500°C. Since most of the PAA pyrolysis gas is hydrogen, hydrocarbon cracking and pore plugging are diminished. Moreover, the high char yield of PAA results in useful strength of the remaining char. Preliminary testing shows the PAA char to be smooth and uniform.

As part of current NASA programs for SRM redesign and improved solid propulsion integrity, NASA Marshall Space Flight Center (MSFC) investigated PAA composites fabricated by MSL. The tests included thermophysical characterization of the material at Southern Research Institute[8] and comparative arcjet ablation testing at MSFC. Two exceptionally thick (2-1/2-in.) carbon-fiber-reinforced PAA composite test billets were fabricated in MSL utilizing PAN-precursor T300 woven carbon fabric as the reinforcement. Our fabrication procedures were demonstrated by these thick billets to be

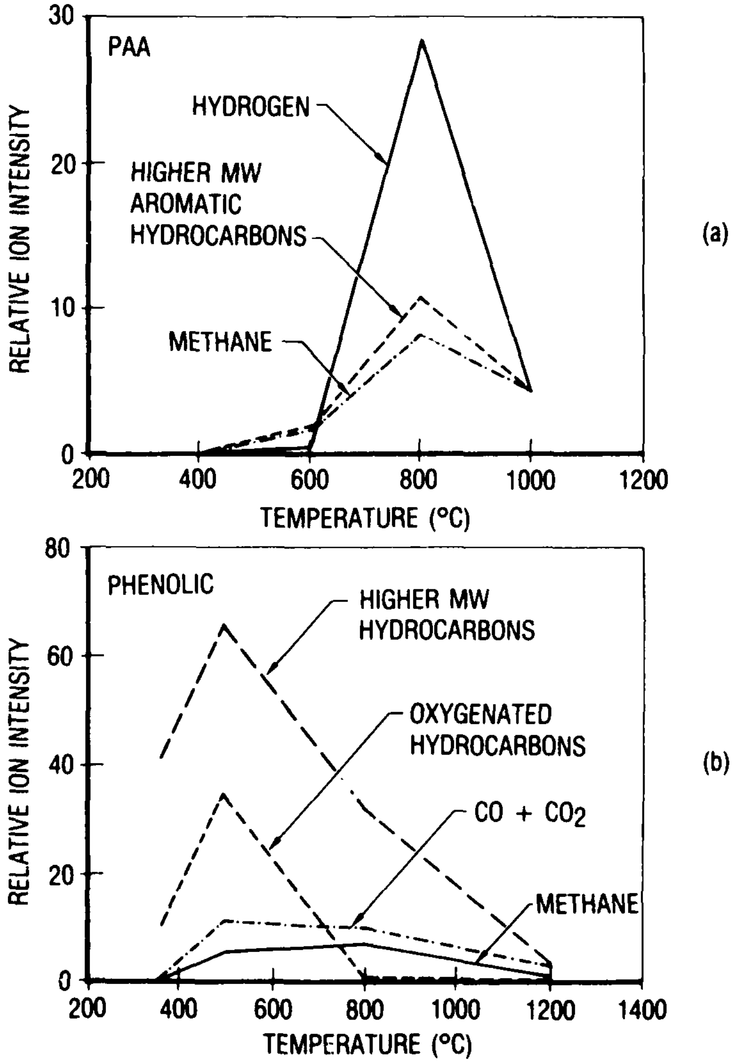

Fig. 9. The composition and relative amounts of gases released during pyrolysis of (a) neat PAA and (b) phenolic resin, measured by gas chromatography mass spectrometry.

suitable for practical nozzle parts and also provided a material whose thermophysical properties could be characterized at both the surface and in the thick part's interior.

NASA performed comparative ablation tests with a CO_2 plasma jet. The ablation tested by this method has been shown to correlate well with recession rates in full-scale motor test firings. The results for PAA composites, as well as for C-Ph (Fig. 10), indicate that PAA is markedly superior--it undergoes less weight loss and erosion--and that PAA data vary much less than those for C-Ph. Also, whereas a large fraction of the C-Ph samples exhibited pocketing, there was no pocketing in any of the PAA samples. NASA currently plans larger scale testing of PAA composites.

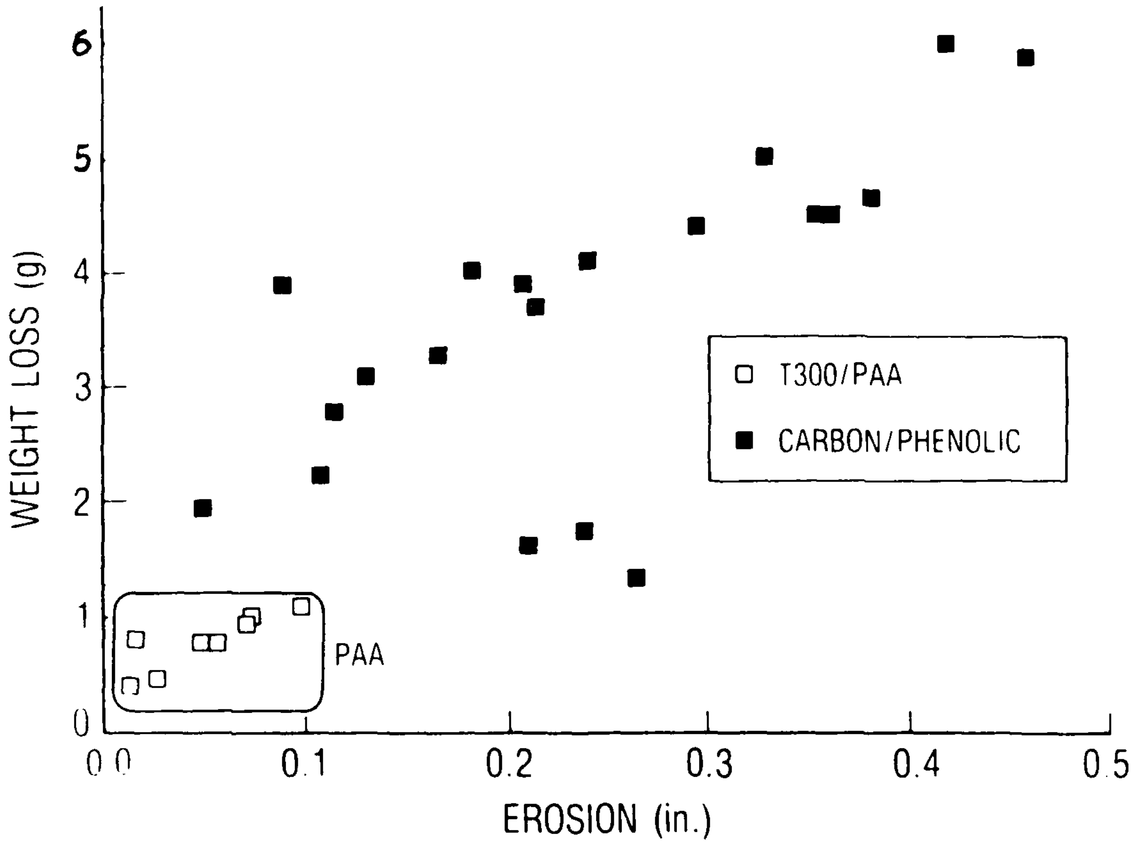

Fig. 10. Results of comparative ablation tests, performed at NASA MFSC, of carbon-fabric-reinforced PAA made in MSL and standard phenolic ablative insulator materials. Specimens were exposed for 10 sec in a high-intensity CO_2 arcjet under standardized conditions.

V. SUMMARY

We have made significant progress in our long-term effort to develop the processing for and to demonstrate the practical application of PAA-based composites. We succeeded in developing a procedure for synthesizing PAA prepolymer, and we established the chemistry of each major processing step required to prepare composite prepreg. The prepreg was used to fabricate large-scale samples and was also demonstrated to be a practical (fast and easy-to-control) precursor for C-C composites. PAA offers promise as a potential low-cost alternative for such composites.

The performance demonstrated in the NASA ablation tests, and NASA's commitment to explore PAA as a part of the next generation of solid boosters, is extremely encouraging. This and other applications of the material will be pursued in the course of our continuing studies of PAA composites.

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LABORATORY OPERATIONS

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Chemistry and Physics Laboratory: Atmospheric chemical reactions, atmospheric optics, light scattering, state-specific chemical reactions and radiative signatures of missile plumes, sensor out-of-field-of-view rejection, applied laser spectroscopy, laser chemistry, laser optoelectronics, solar cell physics, battery electrochemistry, space vacuum and radiation effects on materials, lubrication and surface phenomena, thermionic emission, photosensitive materials and detectors, atomic frequency standards, and environmental chemistry.

Electronics Research Laboratory: Microelectronics, solid-state device physics, compound semiconductors, radiation hardening; electro-optics, quantum electronics, solid-state lasers, optical propagation and communications; microwave semiconductor devices, microwave/millimeter wave measurements, diagnostics and radiometry, microwave/millimeter wave thermionic devices; atomic time and frequency standards; antennas, rf systems, electromagnetic propagation phenomena, space communication systems.

Materials Sciences Laboratory: Development of new materials: metals, alloys, ceramics, polymers and their composites, and new forms of carbon; nondestructive evaluation, component failure analysis and reliability; fracture mechanics and stress corrosion; analysis and evaluation of materials at cryogenic and elevated temperatures as well as in space and enemy-induced environments.

Space Sciences Laboratory: Magnetospheric, auroral and cosmic ray physics, wave-particle interactions, magnetospheric plasma waves; atmospheric and ionospheric physics, density and composition of the upper atmosphere, remote sensing using atmospheric radiation; solar physics, infrared astronomy, infrared signature analysis; effects of solar activity, magnetic storms and nuclear explosions on the earth's atmosphere, ionosphere and magnetosphere; effects of electromagnetic and particulate radiations on space systems; space instrumentation.